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CATIONIC RESIN-MODIFIED SILICA DISPERSION AND ITS PRODUCTION

[Claim(s)]

[Claim 1]It is the dispersion liquid which made silica and cationic resin distribute in a polar solvent, Cationic resin denaturation silica dispersion liquid, wherein mean particle diameter of a silica particle in these dispersion liquid is less than 200 nm and a light scattering index [in / in solids concentration of these dispersion liquid / 1.5 % of the weight] (n value) is 2.0 or more.

[Claim 2]The cationic resin denaturation silica dispersion liquid according to claim 1 whose silica is the silica chosen from wet process silica and dry process silica.

[Claim 3]A manufacturing method of the cationic resin denaturation silica dispersion liquid according to claim 1 carrying out the opposite collision of the mixed liquor produced by mixing silica and cationic resin in a polar solvent above process pressure 300 kgf/cm².

[Claim 4]A manufacturing method of the cationic resin denaturation silica dispersion liquid according to claim 1 passing an orifice under conditions whose differential pressure of an entrance side of an orifice and an outlet side is more than 300 kgf/cm² about mixed liquor produced by mixing silica and cationic resin in a polar solvent.

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to cationic resin denaturation silica dispersion liquid and a manufacturing method for the same. New cationic resin denaturation silica dispersion liquid and manufacturing method useful as a raw material of the still more detailed coating liquid for ink jet recording sheets, etc. are provided.

[0002]

[Description of the Prior Art] Conventionally, silica dispersion liquid are used for the raw material of the coating liquid for ink jet recording sheets.

The silica dispersion liquid which blended the cationized reagent are used for the image concentration of an ink jet recording sheet, and waterproof improvement.

[0003] As silica dispersion liquid used as the above-mentioned coating liquid for ink jet recording sheets, The cation modified colloidal silica which covered the silica surface with the compound of polyvalent metal ion, such as Al ion, to JP,4-19037,B is proposed, The constituent which blended with JP,5-57114,B the cationic resin which contains the 4th class ammonium in the synthetic silica whose diameter of average floc is 0.5-30 micrometers is proposed.

[0004] However, there is a fault that the ink jet recording sheet by which coating was carried out with the coating liquid using the silica indicated to JP,4-19037,B does not have enough performances, such as a water resisting property and color enhancement. Although a water resisting property, color enhancement, etc. improve, since the silica particle is large, the ink jet recording sheet by which coating was carried out with the coating liquid using the constituent indicated to JP,5-57114,B has the fault that surface smoothness and glossiness are low.

[0005] Therefore, in order to solve the above-mentioned problem, the cationized denaturation silica dispersion liquid in which mean particle diameter consists of the silica particle and cationic resin below 200 nm are examined. In this invention, mean particle diameter is volume reference median diameter D₅₀ when it measures with the particle-size-distribution plan of a light scattering diffraction type.

[0006]

[Problem(s) to be Solved by the Invention] However, if mean particle diameter generally mixes cationic resin to the silica dispersion liquid which distributed the silica particle below 200 nm, Even if the silica particle condensed and it carried out re dispersion with the dispersion machine of common use of turbine stator type high velocity revolution

type churning dispersion machines (for example, homogenizer etc.), a colloid mill, an ultrasonic emulsification machine, etc., there was a problem that it did not return to the original dispersion state.

[0007] And by condensation of the above-mentioned silica particle, if the mean particle diameter of silica becomes large, since the penetration of light is barred, a coating layer will become opaque the smooth nature of the surface of a coating layer is not only obtained, but, and the problem that gloss runs short will occur.

[0008] Therefore, the purpose of this invention is to provide the method of manufacturing the cationic resin denaturation silica dispersion liquid and it which condensation which the silica particle described above does not generate and which mean particle diameter becomes from the silica particle and cationic resin below 200 nm.

[0009]

[Means for Solving the Problem] This invention persons mixed liquor produced by mean particle diameter mixing a silica particle and cationic resin below 200 nm in a polar solvent as a result of repeating research wholeheartedly about the above-mentioned problem, When an opposite collision is carried out above process pressure 300 kgf/cm² or differential pressure of an entrance side of an orifice and an outlet side passes an orifice under conditions which are more than 300 kgf/cm², It found out that silica dispersion liquid which carried out re dispersion to the original dispersion state were made again.

[0010] Namely, this invention is the dispersion liquid which made silica and cationic resin distribute in a polar solvent, Mean particle diameter of a silica particle in these dispersion liquid is less than 200 nm, and solids concentration of these dispersion liquid is cationic resin denaturation silica dispersion liquid, wherein a light scattering index (n value) in 1.5 % of the weight is 2.0 or more.

[0011]

[Embodiment of the Invention] The silica in particular used in this invention is not limited. Therefore, in this invention, each publicly known silica, such as wet process silica, dry process silica, and sol-gel method silica, can be used as a raw material.

[0012] The above-mentioned wet process silica has the typical sedimentation method silica formed by mineral acid's neutralizing sodium silicate and depositing silica in a solution, and is also called white carbon. The gel method silica made by neutralizing sodium silicate from acid similarly can also be used. only the drying process which performed filtration and washing after the neutralization reaction is not given -- drying silica -- cake can also be used.

[0013] Generally the above-mentioned dry process silica carries out elevated-temperature hydrolysis of the silicon tetrachlorides in oxygen water matter

flame, is obtained, and is also called fumed silica.

[0014]The above-mentioned sol-gel method silica is obtained by generally hydrolyzing the alkoxide of silicon, such as a tetramethoxy silane and a tetraethoxysilane, in an acid or alkaline hydrous organic solvent.

[0015]In this invention, wet process silica and dry process silica are preferred in the above-mentioned silica.

[0016]In this invention, as cationic resin, If it is resin which dissociates and presents cationicity when it dissolves in water, the resin which it can be used without limitation, and the resin which has the 1st - tertiary amine, or quarternary ammonium salt also in it can use it conveniently, and has quarternary ammonium salt further especially is more preferred.

[0017]If the polar solvent used in this invention is a polar solvent which silica and cationic resin tend to distribute in it, there will be no restriction in particular. Alcohols; ether as this polar solvent, such as water; methanol, ethanol, and isopropyl alcohol; ketone can be illustrated. In the above-mentioned polar solvent, water is preferred. The mixed solvent of water and the above-mentioned polar solvent can also be used.

[0018]In order to raise the stability and dispersibility of a silica particle, a surface-active agent etc. may be added in small quantities to the cationic resin denaturation silica dispersion liquid of this invention in the range which does not spoil the effect of this invention.

[0019]In this invention, the quantity of the silica in cationic resin denaturation silica dispersion liquid, and cationic resin, Although not limited in particular, as for the quantity of the silica in silica dispersion liquid, 8 % of the weight - 50 % of the weight, and also 8 % of the weight - 25 % of the weight are preferred, and the quantity of cationic resin has three to 50 preferred weight section per silica 100 weight section.

[0020]Since the mobility of a slurry will get extremely bad if there is more quantity of the silica in silica dispersion liquid than 50 % of the weight, and mixing with cationic resin becomes difficult, and a large device is needed in order to dry dispersion liquid when less than 8 % of the weight, and also energy cost becomes high on the other hand, it is not desirable.

[0021]If there is less quantity of cationic resin in silica dispersion liquid per silica 100 weight section than three weight sections, it will become uneven balancing it of the surface charge of a silica particle, and a silica particle becomes easy to cause firm condensation. If there is more quantity of cationic resin per silica 100 weight section than 50 weight sections, viscosity will become high and distributed processing will become difficult.

[0022]Not less than +30 mV is still more preferably preferred [the silica particle in the cationic resin denaturation silica dispersion liquid of this invention] for the F-potential used as the index of surface charge not less than +20 mV preferably not less than +10 mV. The water resisting property of inkjet printing paper has an effect, so that F-potential is high. Although the above-mentioned F-potential becomes so high that the mixed amount of cationic resin increases, the margin of increase changes with kinds of cationic resin to mix.

[0023]The cationic resin denaturation silica dispersion liquid of this invention require that the mean particle diameter of the silica particle in these dispersion liquid should be less than 200 nm, and the light scattering index (n value) measured about these dispersion liquid diluted so that solids concentration might be 1.5 % of the weight should be 2.0 or more. If the mean particle diameter of silica is larger than 200 nm and an n value is smaller than 2.0, When it uses as a raw material of the coating liquid for ink jet recording sheets, the penetration of light is barred, a coating layer becomes opaque the smooth nature of the surface of a coating layer is not only obtained, but, and the problem that gloss runs short occurs.

[0024]The above-mentioned light scattering index (n value) is an index of the dispersion state of the silica of dispersion liquid, and this value becomes large as dispersibility improves.

[0025]An n value is Journal of Ceramic Society of Japan 101. [6]It is the value for which 707-712 (1993) was asked according to the method of a statement.

[0026]That is, by measuring the spectrum of the silica dispersion liquid of the range whose wavelength (λ) of light is 460-700 nm using a commercial spectrophotometer, it asks for the absorbance tau, $\log(\lambda)$ and $\log(\tau)$ are plotted, and it asks for inclination (-n) of a straight line with the least square method using a following formula (1).

[0027] $\tau = \alpha \lambda^{-n}$ (1)

(Here, as for tau, an absorbance and alpha show a constant, lambda shows the wavelength of light, and n shows a light scattering index.)

The value which n can take is four or less theoretically.

[0028]If the above-mentioned measuring condition is shown concretely, first, ion exchange water will be filled to a reference cell and a sample cell using 10 mm of light path length's cell, respectively, and zero point proofreading will be performed over the wavelength range of 460-700 nm. Next, the dispersion liquid which diluted dispersion liquid with ion exchange water so that the solids concentration of dispersion liquid might be 1.5% of the weight, and were this diluted by the sample cell are put in, and the

absorbance (τ) of the range of 460-700-nm wavelength (λ) is measured.

[0029]In this invention, there is no exceptional restriction in the method of manufacturing cationic resin denaturation silica dispersion liquid. However, [whether the opposite collision of the mixed liquor produced by mixing silica and cationic resin in a polar solvent as a suitable manufacturing method, for example is carried out above process pressure 300 kgf/cm², and] Or the method of consisting of what the differential pressure of the entrance side of an orifice and an outlet side passes an orifice for under the conditions which are more than 300 kgf/cm² is mentioned.

[0030]The manufacturing method of the mixed liquor of the above-mentioned silica and cationic resin, A method which is not limited but only mixes silica and cationic resin in a polar solvent especially, Turbine Silica dispersion liquid produced by making distribute silica in a polar solvent beforehand using the dispersion machine of common use of stator type high velocity revolution type churning dispersion machines (for example, homogenizer etc.), a colloid mill, an ultrasonic emulsification machine, etc., The method of mixing cationic resin, the method of processing the mixture of silica and cationic resin in a polar solvent using the dispersion machine of the above-mentioned common use, etc. are mentioned.

[0031]The above-mentioned silica used in the manufacturing method of this invention may not be limited in particular for a gestalt, but the thing of which gestalt of a granular material, cake, a slurry, and dispersion liquid may be sufficient as it.

[0032]Also in it, the silica slurry which made the liquid medium distribute a granular material, cake, etc. beforehand, or silica dispersion liquid are preferred, and especially silica dispersion liquid are preferred.

[0033]a consideration of that the raw material of the above-mentioned silica slurry or silica dispersion liquid can be distributed and ground efficiently will not give only the drying process which performed filtration and washing after the neutralization reaction -- the drying silica of wet process silica -- cake is preferred. From a point of the improvement in dispersibility of the cationic resin denaturation silica dispersion liquid obtained, a dry-process-silica granular material is preferred.

[0034]As silica dispersion liquid, although grinding silica dispersion liquid etc. are mentioned, if the effect of this invention is taken into consideration, mean particle diameter is preferred for the grinding silica dispersion liquid below 200 nm also in it. The grinding silica dispersion liquid etc. which various silica specifically ground to the mean particle diameter of less than 200 nm by the method given [the silica slurry currently distributed in the polar solvent] in JP,9-142827,A are mentioned.

[0035]Since the primary particle comprises floc which ten - numbers condensed partly,

especially the grinding silica dispersion liquid obtained by a method given [above-mentioned] in a gazette are excellent in absorbency, and can be conveniently used in the field of an ink jet recording sheet etc.

[0036]Grinding said here means not only the meaning of breaking the silica particle which consists of firm floc but the crack and distribution which unfold condensation of the silica particle which consists of loose floc.

[0037]Pointing out what almost precipitates, when it is neglected, after silica slurry distributed silica in the liquid medium in this invention, silica dispersion liquid refer to what hardly precipitates even if it neglects it, after distributing silica in a liquid medium.

[0038]As for this silica slurry before mixing with cationic resin, or the silica concentration in silica dispersion liquid, 20 or less % of the weight is still more preferably preferred, when using the above-mentioned silica slurry or silica dispersion liquid 30 or less % of the weight preferably 50 or less % of the weight. Since mobility will get extremely bad if 50 % of the weight is exceeded, mixing of cationic resin becomes difficult.

[0039]The mixed liquor produced by mixing silica and cationic resin in a polar solvent in this invention, As a device to carry out an opposite collision above process pressure 300 kgf/cm², or for the differential pressure of the entrance side of an orifice and an outlet side pass an orifice under the conditions which are more than 300 kgf/cm², The device of marketing currently generally called the high voltage homogenizer can use it conveniently. The example of representation of a high voltage homogenizer can be concretely given for Altima IZA made from the trade name; Micro fluidizer and SUGINO machine of illustration then the trade name; nano mizer made from a nano mizer, and the product made from micro fluidics, etc.

[0040]The orifice said here is a mechanism which inserts sheet metal (orifice plate) with which detailed circular hole into a straight pipe, and extracts the channel of a straight pipe rapidly.

[0041]The above-mentioned high voltage homogenizer is a device which consists of the high voltage generating section, opposite collision part, or orifice part which pressurizes stock slurry fundamentally. As a high voltage generating section, high pressure pumping currently generally called the plunger pump is adopted suitably. Although there are various kinds of forms, such as a series type, a 2 ream type, and a 3 ream type, in high pressure pumping, any form is especially employable in this invention without restriction.

[0042]The differential pressure of the entrance side of an orifice and an outlet side in

the case of making the process pressure and the orifice in the case of carrying out an opposite collision pass the mixed liquor produced by mixing silica and cationic resin in a polar solvent in this invention, any -- both -- more than 1200 kgf/cm² is [more than 300 kgf/cm² / more than 800 kgf/cm²] still more preferably desirable preferably.

[0043]As for the collision speed of this mixed liquor in the case of carrying out an opposite collision, it is desirable that it is not less than 150 m/second still more preferably not less than 100 m/second preferably not less than 50 m/second as relative velocity.

[0044]The linear velocity of the polar solvent at the time of passing an orifice is not generally decided, in order to be dependent also on the aperture of an orifice to be used, but it is desirable that it is not less than 150 m/second still more preferably not less than 100 m/second preferably not less than 50 m/second like the collision speed in the case of the above-mentioned opposite collision.

[0045]Also in which method, in order to depend for distributed efficiency on process pressure, distributed efficiency also becomes high, so that process pressure is high. However, if process pressure exceeds 3500 kgf/cm², it will be easy to generate a problem in resistance to pressure, such as piping of high pressure pumping, or the endurance of a device.

[0046]Processing frequency in particular is not restricted but what is necessary is just to determine suitably that the silica dispersion liquid specified by this invention can be obtained also in the method of a gap to describe above. Usually, it is chosen from 1 - the range of ten numbers.

[0047]

[Example]Hereafter, although a reference example, an example, and a comparative example explain this invention concretely, this invention is not restricted at all by these examples.

[0048]The various measurement about silica dispersion liquid was performed as follows.

[0049](Measurement of mean particle diameter) Using the size distribution measuring device (the product made from coal tar, coal tar LS-230) of a light scattering diffraction type, volume reference median diameter D₅₀ was measured and this value was adopted as mean particle diameter. When measuring, the refractive index 1.332 of water (carrier fluid) and the refractive index 1.458 of silica were inputted as a parameter.

[0050](Measurement of viscosity) The silica dispersion liquid 300g were extracted in a 500-cc container, and it stirred for 5 minutes at 20000 rpm using the homogenizer (the product made from a cuttlefish, URUTORATA Lux T-25). Next, after attaching to a 30 ** thermostat for 10 minutes, viscosity was measured on 60-rpm conditions using the

Brookfield viscometer (the TOKIMEC make, BL).

[0051](Measurement of an n value and transmissivity) The visible light absorption spectra of silica dispersion liquid were measured using the spectrophotometer (the Jasco make, Ubest-35 type). First, it filled to the reference cell and the sample cell at ion exchange water using 10 mm of light path length's cell, respectively, and zero point proofreading was performed over the full wave length range. Next, the dispersion liquid which diluted with ion exchange water so that the concentration of silica dispersion liquid might be 1.5% of the weight, and were this diluted by the sample cell were put in, and 241 absorbances (τ) of the range of 460-760-nm wavelength (λ) were measured for every nm. $\log(\lambda)$ and $\log(\tau)$ were plotted and it asked for the slope of a line (-n) with the least square method using the formula (1) mentioned above. Thus, called for n was adopted as a light scattering index.

[0052]Transmissivity (T) was computed with the following formula (2) from the absorbance (τ) with a wavelength of 589 nm (NaD line).

$$[0053] T (\%) = 10^{(2-\tau)} \quad (2)$$

(Measurement of F-potential) The F-potential of the silica particle in silica dispersion liquid was measured using the laser F-potential meter (the Otsuka Electronics make, LEZA-600). First, these dispersion liquid were diluted with a 10 ppm NaCl aqueous solution so that the silica concentration in silica dispersion liquid might be set to 300 ppm, and distributed processing was carried out for 5 minutes by ultrasonic bus. Next, this diluent was put into the measuring cell and F-potential was measured on with the impressed electromotive force 80V, the measurement angle of 20 degrees, and a measurement temperature of 25 ** conditions.

[0054]Reference example (preparation of grinding silica dispersion liquid before mixing with cationic resin)

Commercial sodium silicate and pure water were thrown in so that the solution whose sodium silicate concentration is 5% might be formed into a reaction vessel. After heating this ***** even at 40 ** and counteracting to 50% of a neutralization index using 22wt% sulfuric acid, the temperature of reaction mixture was raised to 95 **. The above-mentioned sulfuric acid was added to this reaction mixture until the neutralization index became 100%. Filtration and washing operation were repeated to the generated silica, and drying cake (silica content 15wt%) was obtained. The specific surface area of the silica produced by making dry this drying cake was 280m²/g.

[0055]The pure water 500g was added to the above-mentioned drying cake 2000g, by agitating with a propeller mixer, preliminary mixing was performed and silica slurry was obtained. The orifice was made to pass the obtained paste state silica slurry 3 times

by process pressure 800 kgf/cm² using a high voltage homogenizer (the product made from a nano mizer; a nano mizer, LA-31), and grinding silica dispersion liquid were obtained. Below, this is called grinding silica dispersion liquid (A). The analysis result is shown in Table 1.

[0056] Reserve mixed liquor was obtained by adding concentration 25wt% of diaryl dimethylammoniumchloride acrylamide copolymer solution [48g of] to 1000 g of example 1 grinding silica dispersion liquid (A) as cationic resin, and agitating with a propeller mixer. Cationic resin denaturation silica dispersion liquid were obtained by making an orifice pass the obtained reserve mixed liquor twice by process pressure 800 kgf/cm² using a high voltage homogenizer (the product made from a nano mizer, nano mizer LA-31). The measurement result about these dispersion liquid is shown in Table 1.

[0057] Cationic resin denaturation silica dispersion liquid were obtained completely like Example 1 except performing an opposite collision for example 2 reserve mixed liquor twice by process pressure 800 kgf/cm² using a high voltage homogenizer (the product made from a nano mizer, nano mizer LA-31). The measurement result about these dispersion liquid is shown in Table 1.

[0058] Cationic resin denaturation silica dispersion liquid were obtained completely like Example 1 except having used concentration 20wt% of diaryl amine salt acid chloride-sulfur dioxide copolymer solution [60g of] as example 3 cationic resin. The measurement result about these dispersion liquid is shown in Table 1.

[0059] Cationic resin denaturation silica dispersion liquid were obtained completely like Example 1 except having used concentration 28wt% of diaryl dimethylammoniumchloride polymer solution [43g of] as example 4 cationic resin. The measurement result about these dispersion liquid is shown in Table 1.

[0060] Instead of using a comparative example 1 high-voltage homogenizer, the homogenizer (the product made from a cuttlefish, URUTORATA Lux T-25) was used, and cationic resin denaturation silica dispersion liquid were obtained like Example 1 except having processed for 10 minutes at 20000 rpm. The measurement result about these dispersion liquid is shown in Table 1.

[0061] Instead of using a comparative example 2 high-voltage homogenizer, the homogenizer (the product made from a cuttlefish, URUTORATA Lux T-25) was used, and cationic resin denaturation silica dispersion liquid were obtained like Example 1 except having processed for 60 minutes at 20000 rpm. The measurement result about these dispersion liquid is shown in Table 1.

[0062] Instead of using a comparative example 3 high-voltage homogenizer, the homogenizer (the product made from a cuttlefish, URUTORATA Lux T-25) was used,

and cationic resin denaturation silica dispersion liquid were obtained like Example 3 except having processed for 10 minutes at 20000 rpm. The measurement result about these dispersion liquid is shown in Table 1.

[0063]Instead of using a comparative example 4 high-voltage homogenizer, the homogenizer (the product made from a cuttlefish, URUTORATA Lux T-25) was used, and cationic resin denaturation silica dispersion liquid were obtained like Example 4 except having processed for 10 minutes at 20000 rpm. The measurement result about these dispersion liquid is shown in Table 1.

[0064]in the sodium silicate and pure water of example 5 marketing, the solution of 5% of sodium silicate concentration is formed into a reaction vessel -- ** -- it supplied like. After heating this solution even at 40 ** and counteracting to 50% of a neutralization index using 22wt% sulfuric acid, the temperature of reaction mixture was raised to 95 **. The above-mentioned sulfuric acid was added until the neutralization index became 100% to this reaction mixture. Filtration and washing operation were repeated to the generated silica, and drying cake (silica content 15wt%) was obtained. The specific surface area of the silica produced by making dry this drying cake was 280m²/g.

[0065]The pure water 200g was added to the above-mentioned drying cake 800g, and silica slurry was obtained by agitating with a propeller mixer. Reserve mixed liquor was obtained by adding concentration 25wt% of diaryl dimethylammoniumchloride acrylamide copolymer solution [48g of] to this silica slurry as cationic resin, and agitating with a propeller mixer. By passing an orifice for this reserve mixed liquor 3 times by process pressure 800 kgf/cm² using a high voltage homogenizer (the product made from a nano mizer, nano mizer LA-31), cationic resin denaturation silica dispersion liquid were obtained. The measurement result about these dispersion liquid is shown in Table 1.

[0066]Instead of using a comparative example 5 high-voltage homogenizer, the homogenizer (the product made from a cuttlefish, URUTORATA Lux T-25) was used, and cationic resin denaturation silica dispersion liquid were obtained like Example 5 except having processed for 10 minutes at 20000 rpm. The measurement result about these dispersion liquid is shown in Table 1.

[0067]

[Table 1]

表1

	n	透過率 (%)	粘度 (mPa·s)	粒子径 (nm)	ゼータ電位 (mV)
粉碎シリカ分散液(A)	2.4	22	10	150	-25
実施例1	2.5	23	50	140	+40
実施例2	2.6	25	50	130	+40
実施例3	2.5	23	100	140	+24
実施例4	2.5	19	53	140	+32
比較例1	1.5	4	820	4000	+47
比較例2	1.6	6	700	3400	+47
比較例3	1.7	10	100	2500	+24
比較例4	0.8	2	190	35000	+39
実施例5	2.4	23	50	150	+41
比較例5	0.8	2	850	35000	+47

All, the cationic resin denaturation silica dispersion liquid of Examples 1-5 were equivalent to grinding silica dispersion liquid (A), or showed the n value and mean particle diameter beyond it so that it might see in Table 1. On the other hand, each cationic resin denaturation silica dispersion liquid of the comparative examples 1-5 had the n value lower than 2.0, and mean particle diameter was not less than 200 nm. The dispersion liquid of the comparative examples 1-5 had low transmissivity again compared with the dispersion liquid of Examples 1-5.

[0068]The pure water 880g was added to 120 g of dry process silica (the Tokuyama make, Reolosil QS30) of example 6 specific surface area [of 300 m]/g, and silica slurry was obtained by carrying out distributed processing with a homogenizer (the product made from a cuttlefish, URUTORATA Lux, T-25). Reserve mixed liquor was obtained by adding concentration 25wt% of diaryl dimethylammoniumchloride acrylamide copolymer solution [48g of] to this silica slurry, and agitating with a propeller mixer as cationic resin, to it. Cationic resin denaturation silica dispersion liquid were obtained by making an orifice pass the obtained reserve mixed liquor once by process pressure 800 kgf/cm² using a high voltage homogenizer (the product made from a nano mizer, nano mizer LA-31). The measurement result about these dispersion liquid is shown in Table 2.

[0069]The pure water 800g was added to 200 g of dry process silica (the Tokuyama make, Reolosil QS30) of example 7 specific surface area [of 300 m]/g, and silica slurry was obtained by carrying out distributed processing with a homogenizer (the product made from a cuttlefish, URUTORATA Lux, T-25). Reserve mixed liquor was obtained by adding concentration 25wt% of diarylmethylamine hydrochloride polymer solution

[concentration 20wt% of / 50g of] to this silica slurry, and agitating with a propeller mixer as cationic resin, to it. Cationic resin denaturation silica dispersion liquid were obtained by making an orifice pass the obtained reserve mixed liquor once by process pressure 800 kgf/cm² using a high voltage homogenizer (the product made from a nano mizer, nano mizer LA-31). The measurement result about these dispersion liquid is shown in Table 2.

[0070]Cationic resin denaturation silica dispersion liquid were obtained completely like Example 7 except having used concentration 50wt% of poly allylamine hydrochloride polymer solution [20g of] as example 8 cationic resin. The measurement result about these dispersion liquid is shown in Table 2.

[0071]Cationic resin denaturation silica dispersion liquid were obtained completely like Example 7 except having used concentration 50wt% of diaryl dimethylammoniumchloride polymer solution [20g of] as example 9 cationic resin. The measurement result about these dispersion liquid is shown in Table 2.

[0072]Instead of using a comparative example 6 high-voltage homogenizer, the homogenizer (the product made from a cuttlefish, URUTORATA Lux T-25) was used, and cationic resin denaturation silica dispersion liquid were obtained like Example 6 except having processed for 10 minutes at 20000 rpm. The measurement result about these dispersion liquid is shown in Table 2.

[0073]Instead of using a comparative example 7 high-voltage homogenizer, the homogenizer (the product made from a cuttlefish, URUTORATA Lux T-25) was used, and cationic resin denaturation silica dispersion liquid were obtained like Example 7 except having processed for 10 minutes at 20000 rpm. The measurement result about these dispersion liquid is shown in Table 2.

[0074]Instead of using a comparative example 8 high-voltage homogenizer, the homogenizer (the product made from a cuttlefish, URUTORATA Lux T-25) was used, and cationic resin denaturation silica dispersion liquid were obtained like Example 8 except having processed for 10 minutes at 20000 rpm. The measurement result about these dispersion liquid is shown in Table 2.

[0075]Instead of using a comparative example 9 high-voltage homogenizer, the homogenizer (the product made from a cuttlefish, URUTORATA Lux T-25) was used, and cationic resin denaturation silica dispersion liquid were obtained like Example 9 except having processed for 10 minutes at 20000 rpm. The measurement result about these dispersion liquid is shown in Table 2.

[0076]

[Table 2]

表2

	n	透過率 (%)	粘度 (mPa·s)	粒子径 (nm)	ゼータ電位 (mV)
実施例6	3.4	62	220	96	+47
実施例7	3.5	63	50	93	+35
実施例8	3.3	60	20	106	+27
実施例9	3.5	65	130	95	+35
比較例6	2.0	39	1500	53870	+49
比較例7	2.7	39	220	6450	+37
比較例8	2.7	39	140	426	+29
比較例9	2.5	31	1600	11670	+36

As it saw in Table 2, mean particle diameter was 200 nm or less, and the n value of each cationic resin denaturation silica dispersion liquid of Examples 6-9 was 2.0 or more. On the other hand, although the n value of each cationic resin denaturation silica dispersion liquid of the comparative examples 6-9 was 2.0 or more, mean particle diameter was not less than 200 nm. The dispersion liquid of the comparative examples 6-9 had low transmissivity again compared with the dispersion liquid of Examples 6-9.

[0077]

[Effect of the Invention] So that I may be understood by the above explanation the cationic resin denaturation silica dispersion liquid of this invention, Although a particulate material is a silica particle with a mean particle diameter of less than 200 nm, Just like [which omits cationized processing] colloidal silica dispersion liquid, since dispersibility is good, it can be conveniently used as a raw material of the coating liquid for inkjet printing paper, and can be further used conveniently also as an inner filler of papers, such as a news print paper, etc.

Abstract:

PROBLEM TO BE SOLVED: To prepare a cationic resin-modified silica dispersion not causing the aggregation of silica particles and comprising silica fine particles having <200 nm average particle diameter and a cationic resin.

SOLUTION: This cationic resin-modified silica dispersion having <200 nm average particle diameter of silica particles and having ≥ 2.0 light scattering index (n) when diluted so as for the concentration of solid content to be 1.5 wt.% is prepared by the opposed collision at ≥ 300 kgf/cm² working pressure or the orifice passage at 300 kgf/cm² differential pressure between the inlet and outlet of the orifice of a mixed liquor prepared by mixing silica with a cationic resin in a polar solvent.